



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 952

Enriched Boric Acid Standard

H ₃ BO ₃ , acidimetric assay, mass fraction	99.97	± 0.02
Absolute abundance ratio, (boron-10/boron-11)	18.80	± 0.02
Boron-10, atom percent	94.949	± 0.005
Boron-11, atom percent	5.051	± 0.005

This Standard Reference Material (SRM) is certified for use as an assay and isotopic standard. The preparation furnished is an enriched boron-10 boric acid. It is slightly contaminated by occluded mother liquor, but the assay value should make it useful by direct weighing for a “spiking” material for boron assays by isotope dilution mass spectrometry, as well as a useful material for the calibration of mass spectrometers.

This lot of boric acid was prepared to ensure material of high purity and homogeneity. As received, it was slightly deficient (approximately 0.01 %) in moisture, but adjusts to a stoichiometric composition after about 30 min exposure to a normal room humidity (approximately 35 % relative humidity). Once adjusted to composition, the material is relatively insensitive (<0.01 %) to moisture changes between 0 % and 60 % relative humidity, and absorbs only about 0.02 % excess moisture at room temperature humidities as high as 90 %. The material cannot be heated as it decomposes with the loss of considerable water.

Assay was by coulometric titration of samples varying in size from 0.2 g to 1.0 g of boric acid, dissolved in 100 mL of a preneutralized solution 1M in KCl and 0.75M in mannitol. The inflection point of the potentiometric curve obtained from measurements with a glass-calomel electrode system was taken as the end point. The pH of the maximum inflection point was taken as the end point. The pH of the maximum inflection point will vary from approximately 7.9 to 8.5 for the range of sample sizes given above, and the titration must, therefore, be conducted in the absence of carbon dioxide or carbonates. The indicated tolerance is at least as large as the 95 % confidence level for a single determination of any sample in the lot of material, and the average essentially indicates a boron-hydrogen ion ratio of 1.0000, since separate examination shows the material contains less than 0.001 % of free strong acid.

The abundance ratio was determined by single filament solid sample mass spectrometry using the Na₂BO₂⁺ ion. Mixtures of known ¹⁰B/¹¹B ratio were prepared from high purity separated isotopes and used as comparison standards. Correction was determined for the ¹⁶O/¹⁷O ratio (¹¹B/¹⁰B ratio -0.00079) by measuring mass 90 using the high purity ¹¹B separated isotope. The indicated tolerance is at least as large as the 95 % confidence limits for a single determination, which includes terms for inhomogeneities in the material as well as analytical error. The atomic weight of the boron, calculated from the absolute abundance ratio and the nuclidic masses 10.0129 and 11.0093, is 10.063.

The technical and support aspects involved in the original preparation, certification, and issuance of this Standard Reference Material were coordinated through the NIST Office of Standard Reference Materials by J.L. Hague. Revision of this certificate was coordinated through the NIST Standard Reference Materials Program by N.M. Trahey.

This Certificate of Analysis has undergone editorial revision to reflect program and editorial changes at NIST and the Department of Commerce. No attempt was made to reevaluate the certificate values or any technical data presented on this certificate.

Willie E. May, Chief
Analytical Chemistry Division

Thomas E. Gills, Director
Office of Measurement Services

Gaithersburg, MD 20899
Certificate Issue Date: 09 November 1999
See Certificate Revision History on Last Page

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of W.R. Shields of the NIST Analytical Chemistry Division.

The material was prepared by the Oak Ridge National Laboratory. The material and the separated isotopes were purified and solutions prepared by K.M. Sappenfield and T.J. Murphy, coulometric titrations were made by G. Marinenko and C.E. Champion, and mass spectrometric measurements were made by E.J. Catanzaro and E.L. Garner all of the NIST Analytical Chemistry Division. Details of the preparations and measurements are available in NBS Special Publication 260-17 [1].

REFERENCE

- [1] NBS Special Publication 260-17, "Standard Reference Materials: Boric Acid; Isotopic and Assay Standard Reference Materials," 70 pp., (February 1970).

Certificate Revision History: 09 November 1999 (editorial revisions); 28 February 1969 (original certificate date).
--

Users of this SRM should ensure that the report in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet <http://ts.nist.gov/srm>.